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# NEW HIGH TEMPERATURE ADDITIVE SYSTEMS FOR PR-143 FLUIDS

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ROLAND E. DOLLE FRANK J. HARSACKY

TECHNICAL REPORT AFML-TR-65-349

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# NEW HIGH TEMPERATURE ADDITIVE SYSTEMS FOR PR-143 FLUIDS

ROLAND E. DOLLE FRANK J. HARSACKY

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### **FOREWORD**

This report was prepared by the Fluid and Lubricant Materials Branch, Nonmetallic Materials Division, Air Force Materials Laboratory, Research and Technology Division. Work was initiated under Project No. 7343, "Aerospace Lubricants," Task No. 734303, "Fluid Lubricants Materials," with Roland E. Dolle and Frank J. Harsacky acting as project engineers.

This report covers work accomplished from 1 October 1964 to 31 January 1965 The manuscript was released by the authors in October 1965 for publication as an RTD technical report.

The additive compounds used in the investigation resulted from in-house efforts of the Polymer Branch, Nonmetallic Materials Division, and were provided to the Fluid and Lubricant Materials Branch as part of a continuing cooperative program between the two Branches. Therefore, the authors wish to acknowledge the contribution of Christ Tamborski and George Baum of the Polymer Branch who were responsible for synthesizing most of the additive materials.

This technical report has been reviewed and is approved.

ROBERT L. ADAMCZAK, Chief Fluid and Lubricant Materials Branch Nonmetallic Materials Division

Air Force Materials Laboratory

### ABSTRACT

Several perfluoroarylmetallic and nonmetallic compounds were highly successful in reducing or eliminating corrosion of ferrous alloys, titanium alloys, and copper immersed in PR-143 at 550° to 600°F under an oxidizing environment. No bulk fluid degradation occurred when the new PR-143 formulations were subjected to oxidative conditions at 600°F in the presence of titanium alloys. Specifically, perfluoroarylphosphine and perfluoroarylphosphine oxide were the most effective of the additive types investigated.

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### INTRODUCTION

Through the cooperative efforts of the Fluid and Lubricant Materials Branch (MANL) and the Organic Chemicals Department of the E. I. du Pont de Nemours and Company during the past two years, a new class of perfluorinated polymers has undergone extensive investigation as lubricants, hydraulic fluids, and heat transfer fluids (References 1 and 2). These new polymeric materials, generally referred to as PR-143, were originally synthesized by du Pont. Due to their high thermal and oxidative stability they are considered the most significant advance in the state of the art of high temperature fluids since the development of the polyphenylethers.

Virtually all of the investigations, both laboratory studies and performance tests, have been with the base or neat fluid, and one major shortcoming has become apparent. At high temperatures (above 500°F) the fluid corrodes ferrous alloys (mild steels, stainless steels, and tool steels), titanium alloys, and copper. In oxidative studies with titanium alloys severe fluid degradation usually resulted.

Certain aluminum alloys are not attacked up to 650°F and some alloys of high nickel content (Inconels, Hastelloys, and Carpenter 20.) are not harmed at 700°F.

For future systems, it may be possible to select only the superalloys during the design stage and thereby eliminate the corrosion problem. This approach, however, is highly restrictive and would exclude the use of the PR-143 fluids in advanced systems of the immediate future. The most promising and the shortest route for obtaining a compatible fluid-metal system is through the use of additives. Preliminary in-house work was begun, employing micro oxidation-corrosion (O-C) tests to evaluate the effectiveness of various available additive compounds in minimizing PR-143 degradation and corrosion of ferrous alloys, titanium alloys, and copper. Reported herein are the results of the formulation study.

### DISCUSSION

GENERAL

The new formulations, prepared from base fluid PR-143, Lot II (ELO-64-20), were evaluated in micro O-C tests according to a procedure outlined previously (Reference 3). However, deviation from the original procedure was made insomuch as the airflow rate was lowered to one liter per hour (original procedure calls for 20 liters per hour). The reduced air rate was necessary due to the volatility of the additive compounds used.

A brief investigation concerning the effectiveness of the new additive materials in PR-143 in a nonoxidizing atmosphere was conducted. The same O-C method was used except dry nitrogen gas was bubbled through the test sample.

The additive candidates chosen were those having perfluoroaryl constituents attached to either a Group IVA (silicon, germanium, tin) or Group VA (nitrogen, phosphorus) atom. Although the exceptional antioxidant ability of various nonfluorinated arylmetallic and non-metallic compounds has been known for some time, the synthesis of their completely fluorinated analogs was accomplished only recently by C. Tamborski and G. Baum of the Polymer Branch, Nonmetallic Materials Division, Air Force Materials Laboratory (Reference 4). Solubility of the new additives in PR-143 due to fluorine substitution coupled with the proven antioxidant characteristics of the basic arylmetallic or nonmetallic structure could offer a successful system for inhibiting PR-143 metal-catalyzed degradation and subsequent metal corrosion.

The following structural formulas represent the perfluoroarylmetallic and nonmetallic compounds examined in PR-143:

$$\left( \left\langle \begin{array}{c} \mathbb{F} \right\rangle \right)_{3}$$

Triperfluorophenylphosphine

$$\left(\left\langle \widehat{F}\right\rangle\right)_{4}$$
-Sn

Tetraperfluorophenyltin

Tetraperiluorophenyigemanium

$$\left(\left\langle \widehat{\mathbb{F}}\right\rangle\right)_{3}$$
  $P \rightarrow 0$ 

Triperfluorophenylphosphine oxide

$$\left(\left\langle F\right\rangle \right)_{4}$$
-\$

Tetraperfluorophenylsilicon

$$\left[-N-P+O-\left(\overline{F}\right)_{2}\right]_{3}$$

Hexaperfluorophenoxytriphosphenitrile

Generally, the PR-143 formulations contained 0.5 weight percent of the above compounds which were completely soluble at that concentration in the base fluid at 212°F. Some of the compounds were soluble at lower temperatures; however, none were soluble in PR-143 at room temperature.

The three nonfluorinated analogs (shown below) which were evaluated for comparison were not soluble in PR-143 even at the test temperature (up to 600°F).

$$\left(\begin{array}{c} \left(\begin{array}{c} \left(\right) \right)} \right) \right) \\ \left( \left(\begin{array}{c} \left(\right) \right) \\ \left( \left(\begin{array}{c} \left(\begin{array}{c} \left(\begin{array}{c} \left(\begin{array}{c} \left(\begin{array}{c} \left(\begin{array}{c} \left(\right) \right) \\ \left( \left(\begin{array}{c} \left(\begin{array}{c} \left(\right) \right) \right) \\ (c \right) \end{array} \right) \\ (c \right) \end{array} \right) \end{array} \right) \end{array} \right) \end{array}} \right) \right) \right) \right) \right) \right) \right)$$

5-Ethyl-5, 10-dihydro-10phenylphenophosphazine Tetraphenyltin

Diphenylbenzylphosphine oxide

Two fluorinated arylamine compounds (Reference 5) were investigated:

Dodecafluoro-phenyl- $\beta$ -naphthylamine

N, N, N', N'-Tetraphenyl-2, 3, 5, 6-tetrafluoro-p-phenylenediamine

### COMPATIBILITY OF FORMULATED PR-143 WITH 410 Stainless Steel

The 400 series stainless steels (ss) were among those most severely attacked in PR-143 base fluids in earlier 600°F micro O-C experiments (Reference 2). It was expected that a significant reduction in the corrosion of a metal from this series, due to additive inhibition, would be clearly indicated when compared with the results for the base fluid. Thus PR-143 formulations containing the aforementioned perfluorinated, partially fluorinated, and non-fluorinated additive compounds were prepared and subjected to micro O-C experiments in the presence of a 410 ss washer at 600°F. The data are summarized in Table I.

As expected, 410 ss was significantly attacked in the base fluid. The metal was corroded moderately accompanied by an appreciable weight increase. In contrast, the 410 ss immersed in the formulations containing triperfluorophenylphosphine and its oxide analog (tests 2 and 3) had no more than a moderate tarnish or discoloration and very little (negligible) weight change. The sample with the perfluorophenoxyphosphonitrile (test 7) also had little corrosive effect on the 410 ss, except that the additive darkened badly and spotted the specimen somewhat.

The other perfluorinated additives were not as successful in inhibiting corrosion, although some improvement was noted (tests 4, 5, and 6). Of the partially fluorinated additives (tests 8 and 9) the dodecassuoro-phenyl- $\beta$ -naphthylamine was best, but degradation of this amine compound was noted.

The nonfluorinated additives charred during the experiments. Only the oxide (test 10) gave significant improvement; however, even in this test, charred additive was deposited on the metal specimen.

It is emphasized that due to the high volatility of the perfluorinated additines at 600°F, much volatilization occurred early in the experiments; nevertheless, the little additive remaining in the fluid sample proved adequate during most of the test period.

The bulk fluid loss due to evaporation was insignificant in all the tests.

COMPATIBILITY OF FORMULATED PR-143 WITH 1006, 1020, 1040, 4130, AND 4140 MILD STEEL

At 550°F (Table II) all five steel specimens corroded in varying degrees in the neat fluid as evidenced by the appearance of the metals and their excessive weight increase (test 13). However in the presence of triperfluorophenylphosphine (test 14) only the 1020 steel was attacked (light corrosion). Negligible weight changes and some discoloration were recorded for the other metals. Bulk fluid change in both tests was negligible.

Similar results were obtained at 600°F (Table II) when the base fluid (test 15) was compared with the phosphine oxide and silicon-containing formulations (tests 16 and 17). At 600°F the effect of the additives was more pronounced than at 550°F since more corrosion occurred in the base fluid at the higher temperature. The silicon-containing additive was less effective than the phosphine oxide at 600°F but, the silicon compound was far more successful in reducing corrosion of the mild steels than it was in reducing corrosion of 410 ss (Table I, test 4). Again the additives volatilized readily, even at 550°F.

COMPATIBILITY OF FORMULATED PR-143 WITH M-1 AND M-2 TOOL STEEL AND 52100 BEARING STEEL

The tool steel and bearing steel specimens immersed in the PR-143 base fluid experienced intense corrosion in the 600°F O-C tests (Table III). Some metal corrosion resulted in the formulation also, particularly with M-1 and M-2 tool steels. However, the weight changes for the M-1 and M-2 specimens in the formulation were considerably less than for the specimens immersed in the base fluid. The condition of the 52100 bearing steel was much improved in the presence of the perfluoroarylphosphine oxide compound. The bulk fluid remain unchanged in both tests.

### COMPATIBILITY OF FORMULATED PR-143 WITH TITANIUM ALLOYS

In the base fluid, both titanium alloys (4% aluminum, 4% manganese alloy and 6% aluminum, 4% vanadium alloy) were attacked as evidenced by their relatively large weight change (Table IV, test 20). It should be noted that the alloy containing aluminum and manganese lost weight and the one containing aluminum and vanadium gained weight. Degradation of the base fluid occurred, as evidenced by the relatively high fluid loss, negative viscosity change, and insoluble formation (white film).

Corrosion (evidenced by excessive weight change) of both titanium alloys was practically eliminated in the formulation containing the phosphine and phosphine oxide compounds (Tests 21 and 22,) although some discoloration or surface dulling of the specimens occurred. The triperfluorophenylphosphine was the most effective of the two compounds.

The related silicon and tin analogs were also successful in suppressing the large weight loss experienced by the alloy containing aluminum and manganese in the base fluid. No significant improvement resulted for the other titanium alloy in the presence of these additives.

The most dramatic effect produced by all of the additive compounds was that the PR-143 fluid did not degrade; the bulk fluid change was small and no white insolubles were formed.

### COMPATIBILITY OF FORMULATED PR-143 WITH COPPER

In the tests with copper at 600°F (Table V) significant corrosion was evident in both the base fluid and formulation. However, the weight loss for the copper specimen was slightly greater in the unformulated sample. Also more spotting of the copper washer occurred in the base fluid. The bulk fluid properties remained unchanged in both tests.

## COMPATIBILITY OF FORMULATED PR-143 WITH MILD STEELS AND TITANIUM ALLOYS UNDER INERT ATMOSPHERE

Earlier studies (Reference 2) have indicated that ferrous alloys and titanium allos corrode in PR-143 base fluid in nitrogen atmosphere (one liter per hour) at 600°F and above after 72 hours. Thus the detrimental effects produced in PR-143-metal systems are not the result of purely oxidative degradation (metal catalyzed), but thermal degradation as well as set aliqued.

Tests for thermal stability (same as the O-C tests except nitrogen is used) where made at 600°F to determine the corrosion-inhibiting effects of triperfluorophenylphosymme in PR-143 in an inert atmosphere, that is, inhibition of metal-catalyzed thermal determination of PR-143 and subsequent metal corrosion. The test duration was limited to 34 hours due to the volatility of the additive. Studies with mild steels and titanium alloys, submarized in Tables VI and VII, showed that neither the steels nor the titanium alloys had significant weight changes in the base fluid or formulation after 24 hours. However, the metals immersed in the formulation had considerably less surface dulling. In the tests with the titanium specimens no bulk fluid degradation resulted with or without the additive.

### CONCLUSIONS AND RECOMMENDATIONS

Certain perfluoroaryl metallic and nonmetallic compounds were highly successful in reducing or eliminating corrosion of ferrous alloys, titanium alloys, and copper in PR-143 fluid above 500°F in air and to some extent in nitrogen atmosphere. Fluid degradation (negative viscosity change and excessive fluid loss) was eliminated in tests with titanium metals.

Partially fluorinated and nonfluorinated aryl compounds containing hydrogen charred during O-C experiments and generally were not successful.

Of the perfluoroaryl compounds examined, triperfluorophenylphosphine and triperfluorophenylphosphine oxide were the most effective in suppressing corrosion. The exceptional performance of these additives in spite of considerable volatilization at 550° to 600°F indicates their effectiveness in very low concentration.

Both solubility and volatility were problems with the prototype additive compounds evaluated. Analogs should be obtained with much improved solubility in PR-143 below room temperature and with lower volatility above 500°F. Improved volatility characteristics alone would allow the additives to be used in PR-143 grease formulations where room temperature solubility is not important if solubility and activation are achieved at the higher operating temperature. Improved volatility and good solubility combined would permit the use of the additives in PR-143 functional fluid and lubricant formulations for operation at the 600° to 700°F level.

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COMPATIBILITY OF FORMULATED PR-143 WITH 410 STAINLESS STEEL TABLE I

i i	Remarks	Fiuid appearance unchanged.	Fluid appearance unchanged.	Fluid appearance unchanged. Much additive volatilized early and darkened slightly.	Fluid appearance unchanged. Much additive volatilized early and darkened slightly.	Fluid appearance unchanged. Additive volatilized early and darkened.	Fluid appearance unchanged. Additive volatilizedearly.	Fluid appearance unchanged. Additive volatilized early.	Fluid appearance unchanged. Additive volatilized early.	Fluid appearance unchanged. Additive volatilized early and darkened badly.
COMOUNTED IN 113 WILL 110 SIMILE 38 SIEEL	Metal $\Delta$ wt. (mg/cm <sup>2</sup> ) and appearance 410 as	+ 0.44 mod. corr., dull	+ 0.49 mod. corr., dull	+ 0.06 mod. tarn., shiny	+0.01 mod. tarn., shiny	+ 0.02 light tarn., shiny	→ 0.22 mod. corr., dull	+ 0.16 very light corr., shiny	+ 0.20 light corr., partly stiny	+ 0.05 light tarnish, some dark spotting.
22.11.77.1	Δ Neut. no., mg KOH/g	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
. 1	Δ Visc. @ 100° F.	+0.6	-0.3	41.3	+ 1.6	-1.9	+ 0.7	+ 1.9	+ 1.6	+ 0.7
	Fluid loss,	6.0	8.0	1.0	0.8	0.5	0.5	0.7	0.7	0.5
	Additive in PR-143	None	None	0.5 wt.% triperfluorophenyl- phosphine	0.5 wt.% triperfluorophenyl- phosphine	0.5 wt.% triperfluorophenyl- phosphine oxide	0.5 wt. % tetraperfluoro- phenylsilicon	0.5 wt.% tetraperfluoro- phenyltin	0.5 wt. % tetraperfluoro- phenylgermanium	0.5 wt.% perfluorophenoxy- triphosphonitrile
	Test No.	1	110	0	Ω2	ო	₩	ເກ	ဖ	2

COMPATIBILITY OF FORMULATED PR-143 WITH 410 STAINLESS STEEL (CONTINUED) TABLE I

<u> </u>	والمراجعة والمساور المراجعة والمراجعة والمراجعة والمراجعة والمراجعة والمراجعة والمراجعة والمساور والمساور					
	Addition in DE-143	Fluid loss,	Δ Visc. @ 100 F.	Neut., no.,	Metal ∆wt. (mg/cm²) and appearance	Remarks
		2	2	0 /2 22 3	22	
	0.5 wt. & doderafiuoro- phenyl- $eta$ -naphthylamine	0.7	۴. ۲. ۵. ۴	0.0	+ 0.10 light corr., dull	Fluid appearance un- changed. Red crystal- line solids collected at top of O-C tube.
	0.5 wt. %, N, N, N', N'- tetrcphenyl-2, 3, 5, 6- tetrafluoro-p-phenylene- Hamine	0.0	+ 0.4	0,0	+ 0.29 smooth dark brown finish, some depos- its	Fiuid cloudy and slightly yellowed. Additive volatilized with some charring.
	0.5 wt.% di,henylbenzyl- phr sphine oxide	0.3	+ 3.3	0.0	+ 0.10 mod. tarn., some varnish b.	Fluid appearance unchanged. Additive volatilized early and charred slightly.
	0.5 wt.% 5-ethyl-5,10- dihydro-10-phenyl- phenophosphazine	£.,	+ 0°3	0.0	+ 0.60 hard dark varnish	Fluid appearance un- changed, Much addi- tive charred,
	0.5 wt.% tetraphenyltin	0.7	+ 4.5	0.0	+ 0.34 mod. corr.	Fluid appearance unchanged, Some additive charred.

Micro O-C test conditions: 20 ml sample, one liver/hr dry air, 60° F, 24-hour duration, overboard, one metal specimen

. The suffix "D" indicates a duplicate test.

Light corrosion was observed in the imperfections on the metal surface. The imperfections were estimated to constitute less than one percent of the total surface area.

TABLE II

COMPATIBILITY OF FORMULATED PR-143 WITH 1006, 1020, 1040, 4130,

Remarks	Fluid appearance unchanged.	Fluid appearance unchanged, Addi- tive volatilized early.	Fluid appearance unchanged.	Fluid appearance unchanged.	Fluid appearance unchanged. Additive volatilized early and dark-ened.
earance 4140 steel	+0.27 light corr.,	+0.02 light tarn.,	+2.18 heavy corr., dull	+ 1.78 heavy corr., dull	+0.06 mcd. tarn., shiny b
and arr 4130 steel	+0.45 mod. corr.,	+0.02 mod. tarn.,	+1.06 mod. corr.,	+1.10 mod. corr.,	+0.30 mod. tarn., shiny b
$g/cm^2$ ) 1040 steel	+0.49 mod. corr.,	+ 0.05 light tarn, shiny	+0.98 mod. corr.,	+0.95 mod. corr.,	+0.12 mod. tarn., shiny b
\		+0.32 light garify shiny	+ 0.24 light corr., partly shiny	+0.35 light corr., partly shiny	+0.12 light tarn., shiny b
	+0.19 light corr.,	+0.04 mod. tarn, shiny	+0.63 mod. corr., dull	+0.34 mod. corr.,	+0.09 mod. tarn., shiry
Neut. no., mg KOH/g	0.0	0.0	0.0	0.0	0 0
ΔVisc. © 100° F,	-1.0	+ 0.6	+ 0.8	0.0	-0.1
Fluid loss,	0.2	0.5	9.0	0.7	0.4
Test dura- tion, hrs	48	48	22	42	42
Test temp.,	550	550	009	600	009
Additive in PR-143	None	0.25 wt. % tri- perfluoro- phenylphos- phine	None	None	0.5 wt. % trf- perfluoro- phenylphos- phine oxide
Test No.	13	14	15	15D	16
	Additive in temp., then, the $\alpha$ is a finite in temp., then, the $\alpha$ is a fixed steel ste	Additive in temp., tion, loss, $\begin{pmatrix} \Delta Visc. & \Delta \\ Fluid & \Delta Visc. \end{pmatrix}$ Neut. no., $\begin{pmatrix} Metal \Delta wt. (mg/cm^2) & and argaearance \\ 1006 & 1020 & 1040 & 4130 & 4140 \\ FR-143 & F & hrs & \% & \% & mg KOH/g & steel & s$	Additive in temp., tion, loss, @ 100°F, Neut. no., PR-143	Additive in temp., tion, 10ss, @ 100°F, Neut. no., 1006  PR-143  None 550 48 0.2 -1.0 0.0 +0.19 +0.55 +0.49 +0.45 +0.27  None 550 48 0.2 -1.0 0.0 +0.19 +0.55 +0.49 +0.45 +0.27  Perfluctor- phenylphos- None 600 24 0.8 +0.8 0.0 +0.63 +0.24 +0.98 +1.06 +1.06  None 600 24 0.8 +0.8 0.0 +0.63 +0.24 +0.98 +1.06 +2.18  None 600 24 0.8 +0.8 0.0 +0.63 +0.24 +0.98 +1.06 +2.18  Metal/Avt. (mg/cm²/cm²/cm²/cm²/cm²/cm²/cm²/cm²/cm²/cm²	Additive in temp., tion, lose, @ 100°F, Neut. no., 1006 1020 1040 4130 4140  PR-143  None 550 48 0.2 -1.0 0.0 +0.19 +0.55 +0.49 +0.45 +0.27  None 600 24 0.8 +0.8 0.0 +0.03 +0.03 +0.08 +1.06 +2.18  None 600 24 0.7 0.0 0.0 +0.34 +0.35 +0.98 +1.06 +2.18  None 600 24 0.7 0.0 0.0 +0.34 +0.35 +0.98 +1.06 +2.18  None 600 24 0.7 0.0 0.0 +0.34 +0.35 +0.98 +1.06 +2.18  Shipp parity dull dull dull dull dull dull dull dul

TABLE II

COMPATIBILITY OF FORMULATED PR-143 WITH 1006, 1020, 1040, 4130 AND 4140 MILD STEELS (CONTINUED)

1	t
Remarks	Fluid appearance unchanged, Additive volatilized early and darkened.
earance 4140 steel	+0.07 +0.14 +0.12 +0.07 +0.15 F mod. mod. utarn., tarn., t
and app 4130 steel	+0.07 mod. tarn., shiny b
1040 1040 steel	+0.12 mod. tarn., shiny b
∆wt. (m 1020 steel	+0.14 light tarn., partly shiny b
Metal 1006 steel	+0.07 mod. tarn., shiny
	0.0
∆V1sc. @ 10° F, %	+ 2, 8
Fluid loss,	6.7
Test dura- temp., tion,	2
Test temp.,	009
Additive in PR-143	0,5 wt. % tet- raperfluoro- phenyleilicon
Test No.	17

Micro O-C test conditions: 20 ml sample, one liter/ar dry air, overboard, five metal specimens.

a. The suffix "D" indicates a duplicate test.

Light corrosion was observed in the imperfections on the metal surface. The imperfections were estimated to constitute less than one percent of the total surface area.

TABLE III

COMPATIBILITY OF FORMULATED PR-143 WITH M-1 AND M-2 TOOL STEEL AND 52100 BEARING STEEL

		Fluid	△ Visc.	۵		Metal $\triangle$ wt. $(mg/cm^2)$ and appearance	) and	
Test No.	Additive in PR-143	loss,	@ 100° F,	, @ 10° F, Neut. no., % mg KOH/g	M-1 tool stæel	M-2 tool steel	52100 bearing steel	Kemarks
18	None	0.5	+ 1,3	0.0	+ 0.89 heavy corr. a.	+ 2, 18 heavy corr. a.	+ 1.34 heavy corr.,	Fluid appearance unchanged.
19	0.5 wt.% triper- fluorophenyl- phosphine oxide	0.5	6 0 +	0.0	+ 0.22 light corr., dull	+ 0, 54 mcd, corr., dull	+ 0, 14 dark tarn. b.	Fluid appearance unchanged, Addi- tive volatilized early and darkened slightly.

Micro O-C test conditions: 20 ml sample, one liter/hr dry air, 600° F, 24-hour duration, overboard, three metal speci-

a. Metal had a gray-black finish.

Light corrosion was observed in the imperfections on the metal surface. The imperfections were estimated to constitute less than one percent of the total surface area.

COMPATIBILITY OF FORMIT, ATEN DR-143 WITH TITANTIM ALLONG TABLE IV

	ì		1	T		Τ	Γ	Γ	
Z C		Remarks	Fluid appearance un- changed. White film formed on O-C tube.	Fluid appearance un- changed. White film formed on O-C tube.	Fluid appearance uncl. aged. Much addi- the volatilizedearly.	Fluid appearance un- changed. Additive volatilized early and darkened slightly.	Fluid appearance un- changed. Additive volatilized early.	Fluid appearance unchanged. Additive volatilized early.	Micro O-C Test Conditions: 20 ml samule, one liter/hr dry sir. 600° F. 24hour duration, everyoard, two metal specimens
TEANIUM ALLO	Metal $\triangle$ wt. (mg/cm <sup>2</sup> ) and appearance	titanium (6% Al, 4% V)	+ 0, 18 d	+ 0, 17 d	+ 0.06 dark tarn., some bluing	p q q	+ 0, 11 d	+ 0,18 d	r diretton, overh
COMPATIBILITY OF FORMULAIED FR-143 WITH TITANIUM ALLOYS	Metal∆ wt. and app	titanium (4% Al, 4% Mn)	-0.35 mod. corr. b	-0.35 mod. corr. b	+ 0.07 dark tarn., some graying	p q +0*0e	+ 0°06 d	+ 0°02 d	600° F. 24hou
AMOLAIED	A Neut. No.,	та кон/в	0.0	0.0	0°0	0.0	0.0	0.0	ter/hr dry afr
	Δ Visc.	@ 100° F. %	-14.6	-14.7	+ 1.0	+ 0.1	+ 1.0	-0.3	mule, one 11
במושא	Fluid	loss, %	6.6	7.1	1.0	0.5	1.2	1,5	.0 ml sa
WOO.		Additive in PR-143	None	None	0.5 wt. % triper- fluorophenylphos- phine	0.5 wt.% triper- fluorophenylphos- phine oxide	0.5 wt.% tetraper- fluorophenylsflicon	0.5 wt.% tetraper- fluorophenyltin	O-C Test Conditions: 2
	Test	o a	20	20D	21	22	23	22	Micro

Test Conditions: 20 ml sample, one liter/hr dry air, 600° F, 24-hour duration, overboard, two metal specimens.

The suffix "D" indicates a duplicate test. Although corrosion was evident from the metal weight loss, a smooth, gray-brown finish was observed on the Graying appearance may be due to some initial corrosion. Metal had a smooth, gray-brown finish. metal specimen. ಲೆ ಕ

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TABLE V COMPATIBILITY OF FORMULATED PR-143 WITH COPPER

Remarks	Fluid appearance unchanged.	Fluid appearance unchanged. Additive volatilized early and darkened slightly.	, overboard, one metal
Metal △wt. (mg/cm²) and appearance copper	-0.68 light spotting, dull	-0.51 very light spotting, dull	ple, one liter/hr dry air, 600° F, 24-hour duration, overboard, one metal
△ Neut. no., mg KOH/g	0.0	0.0	/hr dry air,
ΔV18c. @ 100° F. %	+ 0.6	+ 0.3	de, one liter
Flutd loss, %	0.3	0, 5	20 ml sami specimen.
Additive in PR-143	None	6.5 wt.% triperfluoro- 0.5 phenylphosphine	Micro O-C test conditions: 20 ml sami specimen.
Test No.	25	26	Miero C

COMPATIBILITY OF FORMULATED PR-143 WITH MILD STEELS UNDER INERT ATMOSPHERE TABLE VI

Remarks	Fluid appearance unchanged.	Fluid very slightly yellowed. Additive volatilized early.	20 ml sample, one liter/hr dry nitrogen, 60° F, 24-hour duration, overboard, five metal specimens.
$ \begin{array}{c ccccccccccccccccccccccccccccccccccc$	+ 0.05 light tarn., dull	-0.02 mod. tarn., shiny	hour dura
and ap 4130 steel	+0.06 +0.06 +0.03 +0.02 +0.05 light mod. light light light tarn., shiny	0.00 +0.01 -0.03 -0.02 light mod. mod. mod. tarn., tarn., tarn., tarn., partly shiny shiny shiny	F, 24-1
12/cm <sup>2</sup> ) and ap 1040 4130 steel steel	+0.03 light tarn., partly shiny	0.00 +0.01 light mod. corr., tarn., partly shiny	009 'ue
1020 steel	+0.06 mod. corr.,	ŧ	nitroge
Metal 2 1006 steel	+0.06 light tarn., partly shiny	+0.02 mod. tarn., shiny	/hr dry
$\begin{array}{c c} \Delta & \underline{\text{Metal $\triangle$wt. (II.}}\\ \text{Neut. no., } 1006 & 1020\\ \text{mg KOH/g steel} & \text{steel} \end{array}$	0.0	0.0	le, one liter pecimens.
ΔVisc. @ 100° F,	-0.3	+1.6	20 ml sample, one lit five metal specimens.
Fluid loss,	0.0	0.3	
Additive in PR-143	None	0.5 wt.% triperfluoro- phenylphosphine	Micro thermal stability test conditions:
Test No.	27	88	Micro the

COMPATIBILITY OF FORMULATED PR-143 WITH TITANIUM ALLOYS UNDER INERT ATMOSPHERE TABLE VII

mg/cm <sup>2</sup> )	titanium titanium (4% Al, 4% Mn) (6% Al, 4% V) Remarks	6.00 Fluid appearance unsmooth gray changed.	-0.0l Fluid very slightly light tarn., yellowed. Additive shiny volatilized early.	20 ml sample, one liter/hr dry nitrogen, 600° F, 24-hour duration, overboard, two metal specimens.
Metal $\Delta$ wt. $(mg/cm^2)$	titanium (4% Al, 4% Mn)	+ 0.05 smooth gray finish, dull	+ 0, 02 -0. 0l some graying, light tarn., partly shiny shiny	hr dry nitrogen
\[ \sqrt{\frac{1}{2}} \]	mg KOH/g	0.0	0.0	e, one liter/ ecimens.
V 246.5	@ 10° F,	+ 0.3	0.0	20 ml sample, one lit two metal specimens.
7	lose,	0.3	0.0	nditions:
	Additive in PR-143	Мопе	0.5 wt.% triperfluoro- phenylphosphine	Micro thermal stability test conditions:
	Test No.	53	30	Micro

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KET WORDS	ROLE	WT	ROLE	wT	ROLE	WT
High Temperature Lubricants						
High Temperature Additives						
Formulated PR-143-Type Fluids						
Oxidation-Corrosion		•	<u> </u>			
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